

REPORT ON STANDARDIZATION OF MICROCHEMICAL METHODS

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The Referees on microchemical methods of analyses have continued their collaborative studies this year. The work on methods for the determination of bromine and chlorine, begun in 1951, showed that the Carius and catalytic methods were equally precise and that they were to be preferred to the Parr bomb method. Based upon the data, details, and modifications of these methods employed by the 1951 collaborators, a tentative procedure was developed for each method, and these have been tested collaboratively this year.

Likewise, the collaborative study of the sulfur method is a continuation of that begun in 1951, in which the collaborative results showed a strong preference for the Carius and catalytic methods, both of which were more precise than the Parr bomb method. This year's study has attempted to determine whether either or both of these preferred methods is sufficiently accurate and precise for adoption as an official method.

Also included in the referee collaborative studies is the Dumas method for nitrogen. Initial work on this method was reported in 1949 in which report it was shown that a minimum temperature of 650°C. was required. An evaluation of the effects of a number of variables in the method still remained to be made. This has been done in the current studies.

The 1952 collaborative studies on microchemical methods have led to the following recommendations†:

(1) That the Carius method for bromine and chlorine as used in the 1952 collaborative studies be adopted, first action.

(2) That further studies of the effect of the temperature of the long burner and of the absorbents in the catalytic combustion method for bromine and chlorine should be made.

(3) That the Carius and catalytic combustion methods as described in the 1952 report on the determination of sulfur be adopted, first action.

(4) That the titrimetric method for determining sulfate formed by either of the two combustion procedures for sulfur be adopted, first action.

(5) That further collaborative work should be done to improve the precision of the sulfur gravimetric method which is required for samples containing phosphorus.

(6) That further collaborative work be done to test the method which will be developed from the results of the 1952 studies of the Dumas procedure.

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† For report of Subcommittee C and action of the Association, see *This Journal*, 36, 58 (1953).